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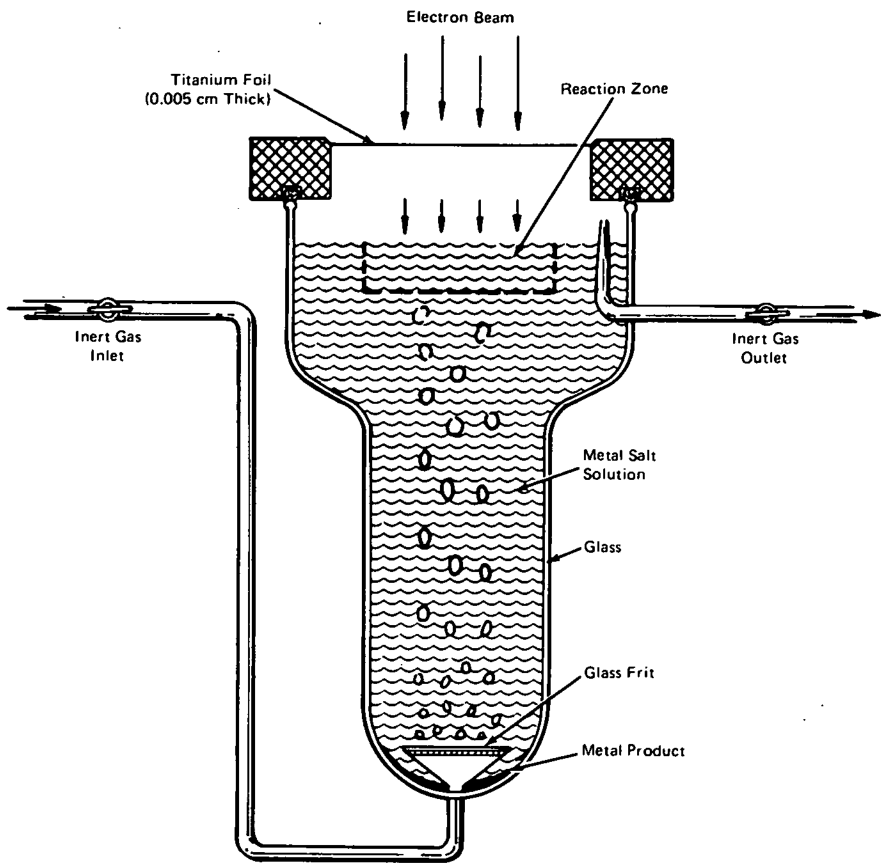
NASA TECH BRIEF

Lewis Research Center

Electron Beam Chemistry Produces High Purity Metals



Production of Metals and Compounds by Radiation Chemistry

The use of radiation chemistry as a practical tool for inorganic preparations is being developed. Most of the effort has involved the deposition of metals by irradiation of their aqueous metal salt solutions with high energy (2 MeV) electrons. The active species in irradiated water are solvated electrons ($e^-_{solvated}$), atomic hydrogen (H·), and hydroxyl radicals (OH·). Addition of certain organic compounds such as primary alcohols generate, by scavenging oxidizing OH·, a reducing medium composed of $e^-_{solvated}$ and H·. These reducing species then reduce the metal ion in solution to deposit the free metal.

The irradiation of the solution is accomplished in the reaction vessel shown in the figure, although other geometries will, of course, occur to practitioners. The titanium foil serves as the inlet window for the vertical electron beam from a linear accelerator. An inactive gas, He, Ar or N_2, passing through a glass frit at the base of the vessel, agitates the liquid through a reaction zone about one centimeter deep (range of penetration of

(continued overleaf)

2 MeV electrons) and about 4 centimeters in diameter (width of beam). The gas then exhausts through the outlet tube.

The features of the radiochemical technique that lend themselves to the preparation of high purity materials are as follows:

1. Low temperatures may be employed thereby minimizing contamination such as that due to diffusion of container material into the product.
2. A wide variety of liquid systems may be used for a particular preparation.
3. The product is deposited as a precipitate thereby affording effective separation of product from reactants.

In a typical experiment, an aqueous solution containing 0.2 molar copper (II) sulfate, 0.05 molar sulfuric acid and 1.0 molar methanol (OH· scavenger) was irradiated with 2 MeV electrons and 20 microamperes beam current. This procedure produced finely divided spectrographic grade copper (crystal size down to 0.1 micron) at a rate of about 50 grams per kW-h beam energy. Ten other metals have been prepared by this method. They are silver, zinc, cadmium, thallium, tin, lead, antimony, cobalt, nickel, and palladium.

The reducing species $e^-_{solvated}$ and H· can also be generated by irradiating certain organic liquids; thus metals can be deposited from nonaqueous media. Antimony and zinc have been precipitated by irradiation of their halide solutions in dry polar organic liquids (e.g., primary alcohols).

Metals may also be deposited by the radiolytic decomposition of alkyl and aryl metal compounds. For example, finely divided lead forms on irradiating a solution of lead tetramethyl in heptane. Likewise, bismuth is radiochemically deposited from a solution of triphenylbismuth in dibutyl ether. These systems would allow the radiolytic preparation of the more electropositive metals such as aluminum and lithium.

Preparations by radiochemical techniques are not confined to the deposition of metals, but can also be used to synthesize pure compounds. Pure anhydrous copper (I) bromide and iron (II) chloride have been prepared in good yield by irradiation of copper (II) bromide and iron (III) chloride, respectively, in organic media.

Theoretical considerations and preliminary experimentation indicate that radiation of considerably less than 2 MeV energy may be used in the radiochemical preparative method; thus a relatively unsophisticated electron source would suffice.

Notes:

1. The following documentation may be obtained from:
   National Technical Information Service
   Springfield, Virginia 22151
   Single document price $3.00
   (or microfiche $0.95)

   Reference: NASA TM-X-67982 (N72-14142), Use of Radiation in Preparative Chemistry
2. Technical questions may be directed to:
   Technology Utilization Officer
   Lewis Research Center
   21000 Brookpark Road
   Cleveland, Ohio 44135
   Reference: B72-10439

Patent status:

Inquiries about obtaining rights for the commercial use of this invention may be made to:

Patent Counsel
Mail Stop 500-311
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135

Source: Warren H. Philipp, Charles E. May,
Stanley J. Marsik, and Robert A. Lad
Lewis Research Center
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